

CLAIMS

1. Process for preparing a composite material comprising an electrode active compound of formula $A_aD_dM_mZ_zO_oN_nF_f$ in which:
- A is an alkali metal,
 - D is chosen from alkaline earth metals and elements of column III of the Periodic Table of Elements, with the exclusion of B,
 - M is a transition metal or a mixture of transition metals,
 - Z is a non-metal chosen from S, Se, P, As, Si, Ge, Sn and B,
 - O is oxygen, N is nitrogen and F is fluorine,
 - a, d, m, z, o, n and f are real numbers greater than or equal to 0 and are chosen so as to ensure electroneutrality;
- and an electronically conducting compound such as carbon;
- in which a homogeneous mixed precursor containing all the elements A, D, M, Z, O, N and F forming the electrode active compound and also one or more organic and/or organometallic compounds are thermally decomposed, in a short period of time, so as to obtain the composite material.
2. Process according to Claim 1, in which said organic and/or organometallic compound(s) is (are) carbon-based compounds preferably comprising a predominant (major) atomic proportion of carbon.

3. Process according to Claim 1, in which A is chosen from Li, Na and K, and mixtures thereof.

5 4. Process according to any one of Claims 1 and 2, in which D is chosen from Mg, Al and Ga, and mixtures thereof.

10 5. Process according to any one of the preceding claims, in which M is chosen from Fe, Ni, Co, Mn, V, Mo, Nb, W and Ti, and mixtures thereof.

15 6. Process according to any one of the preceding claims, in which A is Li or Na, and the electrode active compound is a lithium insertion compound or a sodium insertion compound, such as LiFePO_4 , LiFeBO_3 or NaFeBO_3 .

20 7. Process according to any one of the preceding claims, in which the final percentage by mass of electronically conducting compound such as the carbon in the composite material is from 0.1% to 55%, preferably from 0.2% to 15%.

25 8. Process according to any one of the preceding claims, in which the thermal decomposition of the homogeneous mixed precursor is carried out under vacuum.

30 9. Process according to any one of Claims 1 to 7, in which the thermal decomposition of the

homogeneous mixed precursor is carried out in a controlled atmosphere.

10. Process according to Claim 9, in which
5 the controlled atmosphere is an inert or slightly reducing atmosphere.

11. Process according to any one of the preceding claims, in which the thermal decomposition of
10 the homogeneous mixed precursor is carried out at a temperature of less than 900°C, preferably less than or equal to 800°C, and more preferably less than or equal to 750°C.

12. Process according to Claim 11, in which
15 the thermal decomposition is also carried out at a temperature above 200°C, in particular in the region of 600°C.

13. Process according to any one of the preceding claims, in which the thermal decomposition is carried out over a duration of less than or equal to
20 1 hour, preferably less than or equal to 30 minutes.

14. Process according to Claim 13, in which
25 the thermal decomposition is carried out over a duration of 5 minutes to 1 hour, preferably of 10 minutes to 30 minutes, for example of 15 minutes.

15. Process according to any one of the preceding claims, in which the homogeneous mixed
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precursor compound is prepared by bringing one or more compound(s) containing one or more element(s) chosen from the elements A, D, M, Z, O, N and F forming the electrode active compound into contact, on the molecular scale, with one or more organic and/or organometallic, preferably carbon-based, compound(s) capable of being thermally decomposed, in order to obtain a mixture of said compound(s) containing one or more element(s) chosen from the elements A, D, M, Z, O, N and F and of said organic and/or organometallic compound(s).

16. Process according to Claim 15, in which the respective proportions and the respective compositions of said compound(s) containing one or more element(s) chosen from the elements A, D, M, Z, O, N and F and of said organic and/or organometallic, preferably carbon-based, compound(s), which are thermally decomposable, are chosen so as to observe the proportions of the elements A, D, M, Z, O, N and F, and of the carbon, in the final composite material.

17. Process according to any one of Claims 15 and 16, in which the bringing into contact is carried out in solution, it being possible for said solution to optionally have one or more phases in fine suspension.

18. Process according to any one of Claims 15 and 16, in which the bringing into contact is

carried out by means of a mechanical action, called trituration.

19. Process according to any one of Claims 5 15 to 18, in which, at the end of the bringing into contact, the mixture obtained is dried.

20. Process according to Claim 17, in which the homogeneous mixed precursor is prepared by forming
10 a solution of ions containing the elements to be associated in the compound $A_aD_dM_mZ_zO_oN_nF_f$ and of one or more organic or organometallic, preferably carbon-based, compounds capable of thermally decomposing, which are preferably complexing; and by then very
15 rapidly concentrating said solution so as to fix it and to dry it.

21. Process according to Claim 17, in which the homogeneous mixed precursor is prepared by forming
20 a solution of ions containing the elements to be associated in the compound $A_aD_dM_mZ_zO_oN_nF_f$ and of one or more organic or organometallic, preferably carbon-based, compounds capable of thermally decomposing, and which are preferably complexing, by adding a polyol or
25 a polyamine to said solution in such a way as to carry out a polymerization so as to form a gel, and by drying said gel.

22. Process according to Claim 20 or 21, in
30 which said carbon-based organic compound(s) capable of thermally decomposing, and which is (are) preferably

complexing, is (are) chosen from organic acids containing two acid functions or more, such as oxalic acid, malonic acid, succinic acid, glutaric acid, adipic acid, maleic acid or fumaric acid; alcohol
5 acids, such as glycolic acid, lactic acid, mandelic acid, hydroxyacrylic acid or hydroxybutyric acid; amino acids, such as aminoacetic acid, also called glycine, alanine, leucine, aminopropionic acid, ornithine, lysine or arginine; ketone acids, such as glyoxylic
10 acid, pyruvic acid, ketobutyric acid or levulic acid; acids which are more complicated, bearing two or more acid functions and other alcohol, amine or carbonyl functions, such as malic acid, tartaric acid, citric acid, aconitic acid, citraconic acid, aspartic acid and
15 glutamic acid; and mixtures thereof.

23. Process according to Claim 21, in which said polyol is chosen from glycols, preferably from (C₁ to C₆)alkylene and poly(C₁ to C₆)(alkylene) glycols,
20 such as ethylene glycol and diethylene glycol.

24. Process according to Claim 17, in which the homogeneous mixed precursor is prepared by forming a solution of ions containing the elements to be
25 associated in the compound $A_a D_d M_m Z_z O_o N_n F_f$, and by adding to this solution one or more water-soluble gelling organic compounds, in order to form an organic gel comprising said ions, and then by drying said gel.

30 25. Process according to Claim 24, in which said gelling organic compound(s) is (are) chosen from

(meth)acrylamides, (meth)acrylates and polymerizable carbohydrates, such as starch and saccharides and derivatives thereof.

5 26. Process according to Claim 17, in which the homogeneous mixed precursor is prepared by direct sol-gel polymerization between an alkoxide of the element Z such as silicon, and a generally complexing oxoanion such as $(ZO_4)^{x-}$ or a precursor thereof, in a
10 solution of ions containing the elements A, D and M to be associated in the compound $A_aD_dM_mZ_zO_oN_nF_f$ such that the metals A, D and M are trapped in the network thus formed, and then by drying.

15 27. Process according to any one of Claims 1 to 26, in which the composite material is in the form of a light ash consisting of very fine grains of between 100 and 5000 \AA in size.

20 28. Process according to Claim 27, in which the composite material also has a specific surface area of from 10 to 50 m^2/g .